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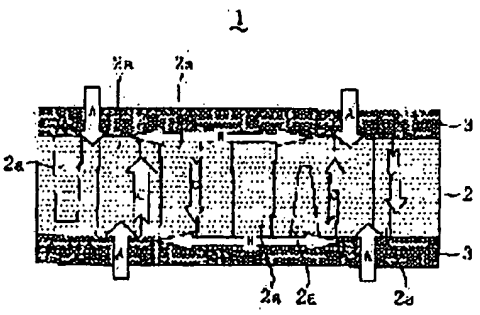
(54) HYDROGEN OCCLUDING LAMINATED STRUCTURE

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a hydrogen occluding laminated structure whose hydrogen discharging temperature is low while holding a high amount of hydrogen to be occluded.

SOLUTION: This hydrogen occluding laminated structure 1 is provided with a hydrogen occluding layer 2 composed of Mg or an Mg based hydrogen occluding alloy whose structure is made into the nano one and a pair of hydrogen transferring layers 3, 3 laminated on both surfaces of the hydrogen occluding layer 2 so as to hold the same, respectively giving catalytic effect capable of dissociating gaseous hydrogen into an atomic state, also diffusing the dissociated hydrogen atoms over the insides and transferring the hydrogen atoms into/ from the hydrogen occluding layer 2.



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CLAIMS

[Claim(s)]
[Claim 1] The hydrogen absorption laminating structure which a laminating is carried out to both the front face so that the hydrogen absorption layer which consists of Mg or Mg system hydrogen storing metal alloy by which nano structuring was carried out, and the above-mentioned hydrogen absorption layer may be pinched, and is made to carry out internal diffusion of the hydrogen atom which discovered and dissociated respectively the catalysis which may make hydrogen gas dissociate in the shape of an atom, and is characterized by having this hydrogen absorption layer and the hydrogen transfer layer of the pair which performs transfer of a hydrogen atom.
[Claim 2] The above-mentioned hydrogen absorption layer is the hydrogen absorption laminating structure according to claim 1 characterized by including a nano columnar crystal.
[Claim 3] The above-mentioned hydrogen absorption layer is the hydrogen absorption laminating structure according to claim 1 characterized by including nano crystal grain.
[Claim 4] The above-mentioned nano columnar crystal is the hydrogen absorption laminating structure according to claim 2 characterized by column width being 50nm or less.
[Claim 5] It is the hydrogen absorption laminating structure according to claim 1 to 4 characterized by at least one side containing a noble-metals element among the hydrogen transfer layers of a top Norikazu pair.

[Translation done.]

JAPANESE [JP,2002-105576,A]

CLAIMS DETAILED DESCRIPTION TECHNICAL FIELD PRIOR ART EFFECT OF THE INVENTION TECHNICAL PROBLEM MEANS EXAMPLE DESCRIPTION OF DRAWINGS DRAWINGS

[Translation done.]

JAPANESE [JP,2002-105576,A]

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[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the hydrogen absorption laminating structure.
[0002]
[Description of the Prior Art] In recent years, hydrogen attracts attention as a clean energy source which the air pollution and the global warming by use of a fossil fuel, such as a fuel oil and a gasoline, are greatly dealt with as an environmental problem, and is replaced with a fossil fuel. And in order to use hydrogen as an energy source, the medium for carrying out occlusion of it is needed.
[0003] As this medium, in order to carry out occlusion of a lot of hydrogen to 7.6 mass % by the shape of bulk (massive), promising ** of Mg is carried out as one of the component elements of the alloy which constitutes hydrogen absorption material or hydrogen absorption material. However, since the hydride (MgH2) is thermodynamically stable, Mg needs an elevated temperature 300 degrees C or more for emission of hydrogen, and this has become a big neck to utilization.
[0004] On the other hand, the hydrogenation property of a metal thin film is studied in order to mainly investigate the behavior of the hydrogen atom in a metal. Although research fundamental also about the hydrogenation property of Mg thin film is made, and it is clear about low temperature-ization of hydrogen desorption temperature being attained by thin-film-izing Mg, the thing with potential sufficient as hydrogen absorption material is not reported until now. In the Pd/Mg laminating thin film created with vacuum deposition, when the hydrogen storage capacity of Mg is as low as 0.5 mass %, specifically, it is reported that Mg emits hydrogen at about 90 degrees C.
[0005] this invention person moreover, by the RF (RF) exchange magnetron sputtering method If a Pd/Mg laminating thin film is created using Ar gas pressure as 0.5-1.0Pa If it hydrogenates as it is and oxidation is prevented, without Mg layer's becoming what was formed of the columnar structure with a column width of 100nm or less which carried out nano structuring, and making atmospheric air touched after forming this It has found out that hydrogen desorption temperature can be made to low-temperature-ize to the 110 degrees C of the minimum abbreviation, maintaining the high hydrogen storage capacity of 2 - 6 mass % in Mg layer. However, the hydrogen storage capacity made into the practical use range turns into 3 mass % and high capacity, and that whose hydrogen desorption temperature is 100 degrees C or less is not still obtained. In addition, Pd layer has the catalysis which may make hydrogen gas dissociate in the shape of an atom, and plays a role of an entrance of the hydrogen atom to Mg layer here.
[0006] Furthermore, the charge of a hydrogen absorption laminated wood which carried out the laminating of the layer which contains 4A group elements, such as Ti whose stable crystal structure is hcp structure, under ordinary temperature and ordinary pressure, and the layer which contains elements, such as Cr whose stable crystal structure is bcc structure, under ordinary temperature and ordinary pressure is indicated by JP,9-59001,A, and it is indicated that the occlusion engine performance of hydrogen in which it excelled by this configuration is discovered. However, the indication about hydrogen desorption temperature is not made by this.

[0007]
[Problem(s) to be Solved by the Invention] The technical problem of this application is to offer the hydrogen absorption laminating structure with low hydrogen desorption temperature, though a high hydrogen storage capacity is held.
[0008]
[Means for Solving the Problem] This invention carries out internal diffusion of the hydrogen atom which discovered and dissociated the catalysis which may make hydrogen gas dissociate the hydrogen absorption layer which consists of Mg or Mg system hydrogen storing metal alloy by which nano structuring was carried out in the shape of an atom, and it carries out a laminating so that it may insert in a hydrogen absorption layer and the hydrogen transfer layer of the pair which delivers and receives a hydrogen atom.
[0009] It is the hydrogen-absorption laminating structure which a laminating is carried out to both the front face so that the hydrogen absorption layer which consists of Mg or Mg system hydrogen storing metal alloy with which nano structuring of this invention was specifically carried out, and the above-mentioned hydrogen absorption layer may be pinched, and is made to carry out internal diffusion of the hydrogen atom which discovered and dissociated the catalysis which may make hydrogen gas dissociate in the shape of an atom, and is respectively characterized by to have this hydrogen absorption layer and the hydrogen transfer layer of the pair which performs transfer of a hydrogen atom.
[0010] Since internal diffusion of the hydrogen atom to which the hydrogen absorption layer which consists of Mg or Mg system hydrogen storing metal alloy by which nano structuring was carried out according to the above-mentioned configuration discovered and dissociated the catalysis which may make hydrogen gas dissociate in the shape of an atom is carried out, and the laminating is carried out so that it may be inserted in a hydrogen absorption layer and the hydrogen transfer layer of the pair which performs transfer of a hydrogen atom, although a high hydrogen storage capacity is held, hydrogen desorption temperature will become low. The following things are presumed although it is not clear about this mechanism.
[0011] First, since the hydrogen absorption layer is arranged for the hydrogen transfer layer which is made to carry out internal diffusion of the hydrogen atom which discovered and dissociated the catalysis which makes hydrogen gas dissociate in the shape of an atom, and is delivered and received between hydrogen absorption layers up and down, respectively, the entrance of the hydrogen atom to a hydrogen absorption layer will be secured widely, and hydrogen absorption and emission of a hydrogen absorption layer will be performed smoothly.
[0012] Moreover, since the hydrogen absorption layer to which the laminating of the hydrogen transfer layer was carried out up and down will also receive cubical expansion and the force to contract in connection with it according to the occlusion and emission of a hydrogen atom in order to contract, cubical expansion and, a hydrogen transfer layer While the occlusion of a hydrogen atom is promoted in response to the force in which the volume of a hydrogen absorption layer expands, at the time of the occlusion of the hydrogen atom of a hydrogen transfer layer, at the time of emission of the hydrogen atom of a hydrogen transfer layer It will be urged to the emission of a hydrogen atom which was carrying out occlusion in response to the force which the volume of a hydrogen absorption layer contracts, and hydrogen absorption and emission of a hydrogen absorption layer will be performed smoothly.
[0013] The amorphous field of hydrogen absorption ability whose atomic arrangement is a non-dense is higher than the crystalline region where atomic arrangement is dense. Furthermore, the sake, By following on expansion and contraction of a crystalline region, and expanding and contracting an amorphous field Then, hydrogen absorption and emission are promoted in response to the operation with an elastic hydrogen atom, namely, generally an amorphous field is considered to be what mainly carries out occlusion of the hydrogen to a crystalline region being what mainly promotes hydrogen absorption and emission. On the other hand, nano structuring is carried out and the hydrogen absorption layer has become what has the continuous change to the random atomic arrangement of an amorphous field from the regular atomic arrangement of a crystalline region. Moreover, since it is thought that the energy

barrier at the time of a hydrogen atom diffusing between both fields mutually is small, the diffusion path of a hydrogen atom is secured greatly and a hydrogen atom can go both fields back and forth smoothly. Expansion and contraction of both fields will receive the operation with an elastic hydrogen atom, and hydrogen absorption and emission will be smoothly performed to coincidence in both fields.

[0014] Here, "nano structure" consists of a crystalline region and an amorphous field (metastable phase with an amorphous phase and short-distance order), and means the structure where these fields consist of NANOMETORU (ten 9th [-] power meter) scales.

[0015] Moreover, "Mg system hydrogen storing metal alloy" means the hydrogen storing metal alloy which used Mg, such as a Mg-nickel system and a Mg-Re (rare earth elements) system, as the base.

[0016] And the thing in which column width contains a nano columnar crystal 100nm or less, then the above-mentioned operation effectiveness will be performed proper, and a hydrogen absorption layer has them. [desirable] In this case, as for a nano columnar crystal, it is more desirable that column width sets to 50nm or less.

[0017] Moreover, a hydrogen absorption layer can acquire the same operation effectiveness as the case where a nano columnar crystal is included also as a thing containing nano crystal grain. In that case, as for the diameter of crystal grain, it is desirable to be referred to as 50nm or less.

[0018] Furthermore, as for at least one side, it is desirable among the hydrogen transfer layers of a pair that noble-metals elements, such as Pd and Pt, are included. A noble-metals element is because the catalyst engine performance which makes hydrogen gas dissociate in the shape of an atom is high. As for especially PdH and Pd which constitutes an about 0.6-PdH hydride in fact, it is desirable for the diffusibility ability in a layer of a hydrogen atom to be superior to other noble metals, and to make it include this in a hydrogen transfer layer.

[0019] Moreover, you may make it include similarly both either of the transition-metals elements which have a peripheral electron into 3d orbits, such as either of the rare earth elements, such as La and Nd, or V, nickel, Cu, or these rare earth elements, and a transition-metals element at least in one side among the hydrogen transfer layers of a pair.

[0020] The above hydrogen absorption laminating structures consider as the three-tiered structure object which consists of a hydrogen transfer layer by which the laminating was carried out so that a hydrogen absorption layer and it may be inserted from the upper and lower sides, and also they are good as the five-layer structure which carried out the laminating of a hydrogen transfer layer and the hydrogen absorption layer by turns, or the seven-layer structure. However, both ****** need to be taken as a hydrogen transfer layer in this case.

[0021] And if coarse grinding of this hydrogen absorption laminating structure is carried out with a laminated structure held and a container is filled up with it, it can be used for a stationary-type or mobile-type hydrogen absorption object.

[0022] Moreover, if nickel porous bodies, such as foaming nickel, are made to support the powder of this hydrogen absorption laminating structure, it can also be used as an electrode of a rechargeable battery. In this case, if the laminating structure is prepared on a nickel substrate, it can use as a negative electrode as it is.

[0023] Furthermore, that by which the hydrogen absorption layer of this hydrogen absorption laminating structure prepared the laminating structure on transparence substrates, such as glass, for example from changing to light impermeability reversibly at the time of light transmission and hydrogen desorption at the time of hydrogen absorption being found out, and being able to realize easily the manifestation of the reversible light transmission nature of the hydrogen absorption layer accompanying hydrogen absorption emission is applicable to a display, a SUICHA bull mirror, a sunroof, etc. However, in order to give high light transmittance in this case, as for a hydrogen transfer layer, it is desirable to make it as thin as possible below 10-nanometer order. In addition, this property is discovered also when a hydrogen absorption layer consists of Mg simple substances.

[0024]

[Effect of the Invention] Since internal diffusion of the hydrogen atom to which the hydrogen absorption layer which consists of Mg or Mg system hydrogen storing metal alloy by which nano structuring was

carried out according to this invention as explained above discovered and dissociated the catalysis which may make hydrogen gas dissociate in the shape of an atom is carried out, and the laminating is carried out so that it may be inserted in a hydrogen absorption layer and the hydrogen transfer layer of the pair which performs transfer of a hydrogen atom, although a high hydrogen storage capacity is held, hydrogen desorption temperature will become low.

[0025]

[Embodiment of the Invention] Hereafter, the operation gestalt of this invention is explained to a detail based on a drawing.

[0026] Drawing 1 shows the configuration of the hydrogen absorption laminating structure concerning the operation gestalt of this invention.

[0027] This hydrogen absorption laminating structure 1 consists of three-tiered structures which consist of a hydrogen absorption layer 2 and hydrogen transfer layers 3 and 3 to which the laminating of that hydrogen absorption layer 2 was carried out up and down.

[0028] The hydrogen absorption layer 2 is constituted by the nano columnar crystals 2a and 2a with a column width of 50nm or less which carried out orientation in the direction of thickness, and Mg simple substance containing -- by which nano structuring was carried out. Mg has the function which carries out occlusion of a lot of hydrogen to 7.6 mass % by the shape of bulk (massive).

[0029] Each hydrogen transfer layer 3 is constituted by Pd simple substance.

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EFFECT OF THE INVENTION

[Effect of the Invention] Since internal diffusion of the hydrogen atom to which the hydrogen absorption layer which consists of Mg or Mg system hydrogen storing metal alloy by which nano structuring was carried out according to this invention as explained above discovered and dissociated the catalysis which may make hydrogen gas dissociate in the shape of an atom is carried out, and the laminating is carried out so that it may be inserted in a hydrogen absorption layer and the hydrogen transfer layer of the pair which performs transfer of a hydrogen atom, although a high hydrogen storage capacity is held, hydrogen desorption temperature will become low.
[0025]
[Embodiment of the Invention] Hereafter, the operation gestalt of this invention is explained to a detail based on a drawing.
[0026] Drawing 1 shows the configuration of the hydrogen absorption laminating structure concerning the operation gestalt of this invention.
[0027] This hydrogen absorption laminating structure 1 consists of three-tiered structures which consist of a hydrogen absorption layer 2 and hydrogen transfer layers 3 and 3 to which the laminating of that hydrogen absorption layer 2 was carried out up and down.
[0028] The hydrogen absorption layer 2 is constituted by the nano columnar crystals 2a and 2a with a column width of 50nm or less which carried out orientation in the direction of thickness, and Mg simple substance containing -- by which nano structuring was carried out. Mg has the function which carries out occlusion of a lot of hydrogen to 7.6 mass % by the shape of bulk (massive).
[0029] Each hydrogen transfer layer 3 is constituted by Pd simple substance. Pd has the function to which internal diffusion of the catalysis which makes hydrogen gas dissociate in the shape of an atom is discovered and carried out.
[0030] The hydrogen absorption mechanism of this hydrogen absorption laminating structure 1 is considered as follows, although it is not clear.
[0031] first, the thing for which it has the admission passage of hydrogen gas up and down as the hydrogen absorption layer 2 is arranged up and down, respectively and the hydrogen transfer layers 3 and 3 show by the arrow heads A and A of drawing 1 , and -- ** -- since it becomes, the entrance of the hydrogen atom to the hydrogen absorption layer 2 will be secured widely, and hydrogen absorption and emission of the hydrogen absorption layer 2 will be performed smoothly.
[0032] The hydrogen transfer layers 3 and 3 respond to the occlusion and emission of a hydrogen atom. Moreover, in order [cubical expansion, in order to contract], Since cubical expansion and the force to contract will be received in the direction which also shows the hydrogen absorption layer 2 to which the laminating of the hydrogen transfer layers 3 and 3 was carried out up and down to the arrow heads B and B of drawing 1 in connection with this At the time of the occlusion of the hydrogen atom of the hydrogen transfer layers 3 and 3 While the occlusion of a hydrogen atom is promoted in response to the force in which the volume of the hydrogen absorption layer 2 expands, at the time of emission of the hydrogen atom of the hydrogen transfer layers 3 and 3 It will be urged to the emission of a hydrogen atom which was carrying out occlusion in response to the force which the volume of the hydrogen

absorption layer 2 contracts, and hydrogen absorption and emission of the hydrogen absorption layer 2 will be performed smoothly.

[0033] Furthermore, nano structuring is carried out and the hydrogen absorption layer 2 has become what has the continuous change to the random atomic arrangement of an amorphous field from the regular atomic arrangement of a crystalline region. moreover, between both fields from it being thought that the energy barrier at the time of a hydrogen atom being spread mutually is small As the arrow heads C and C of drawing 1 and -- show, the diffusion path of a hydrogen atom is secured greatly and a hydrogen atom can go both fields back and forth smoothly. Expansion and contraction of both fields will receive the operation with an elastic hydrogen atom, and hydrogen absorption and emission will be smoothly performed to coincidence in both fields.

[0034] Next, the manufacture approach of this hydrogen absorption laminating structure is explained.

[0035] Drawing 2 shows the configuration of RF exchange magnetron sputtering system used for the manufacture.

[0036] RF exchange magnetron sputtering system is the thing of a configuration of having prepared the coil for inductive-coupling RF plasma discharge above the planar magnetron cathode. According to this equipment, it becomes producible about the thin film which sputtering in a low voltage ambient atmosphere is possible, and are high density and a high grade, and was excellent in smooth nature. Moreover, the membrane formation to a substrate with the formation of a low plasma damage and heat-resistant temperature lower than a long distance between a substrate and a target can be taken and the thickness control in a quartz-resonator type thin film monitor become possible. Furthermore, precision control (a compound, alloy) of a multicomponent film presentation and precision control (multilayers, superlattice) of a laminating period are attained from a plural coincidence spatter being possible. Moreover, about the uniformity of membrane formation, uniform membrane formation is attained from a cathode in large size.

[0037] And sputtering is carried out to the order of Pd, Mg, and Pd on substrates, such as glass, a quartz, and nickel plate, in Ar gas ambient atmosphere using this RF exchange magnetron sputtering system. At this time, sputtering of Mg performs Ar gas pressure as 0.5-1.0Pa. Of this, Mg layer (hydrogen absorption layer) containing a nano columnar crystal with a column width of 50nm or less which carried out orientation by which nano structuring was carried out will be formed in the direction of thickness.

[0038] Since internal diffusion of the hydrogen atom to which the hydrogen absorption layer 2 which consists of Mg simple substance by which nano structuring was carried out according to the hydrogen absorption laminating structure 1 of the above-mentioned configuration discovered and dissociated the catalysis which may make hydrogen gas dissociate in the shape of an atom is carried out, and the laminating is carried out so that it may be inserted in a hydrogen absorption layer and the hydrogen transfer layers 3 and 3 of the pair which consists of Pd simple substance which delivers and receives a hydrogen atom, although a high hydrogen storage capacity is held, hydrogen-desorption temperature will become low.

[0039] And since the hydrogen absorption layer 2 consists of nano columnar crystals 2a and 2a with a column width of 50nm or less and Pd in which, as for the hydrogen transfer layers 3 and 3, -- forms a hydride (PdH) including Mg and the engine performance which diffuses a hydrogen atom in the hydrogen transfer layer 3 and 3 is high, the above-mentioned operation effectiveness becomes what was extremely excellent.

[0040] In addition, although the hydrogen absorption laminating structure 1 was made into the three-tiered structure with the above-mentioned operation gestalt, it is good also as the five-layer structure which is not limited to especially this, used both ****** as the hydrogen transfer layer, and carried out the laminating of a hydrogen transfer layer and the hydrogen absorption layer by turns, or the seven-layer structure. In this case, the hydrogen gas introduced from the hydrogen transfer layer outside both ** is mainly dissociated by the hydrogen atom. When it is advanced and spread in the hydrogen absorption layer [directly under] of it after the dissociated hydrogen atom diffuses the inside of an outermost hydrogen transfer layer, and a hydrogen atom is further advanced and spread in a middle hydrogen transfer layer A hydrogen atom is promptly spread in the whole laminating structure, and it is

presumed that it is that by which a hydrogen atom is efficiently incorporated by the internal hydrogen absorption layer. Moreover, the hydrogen transfer layer is considered that a middle hydrogen transfer layer is what has achieved the function like a pump which it makes a hydrogen atom permeate to an internal hydrogen absorption layer since the diffusion rate of a hydrogen atom is quick rather than a hydrogen absorption layer.

[0041] Moreover, with the above-mentioned operation gestalt, although the hydrogen absorption layer 2 shall contain the nano columnar crystals 2a and 2a and --, it is not limited to especially this and nano crystal grain may be included.

[0042] Moreover, with the above-mentioned operation gestalt, although the hydrogen absorption laminating structure 1 was manufactured by the RF exchange magnetron sputtering method, it is not limited to especially this and you may manufacture in powder metallurgy, such as the rolling-out method.

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EXAMPLE

[Example] (Test evaluation 1)
By each approach of the magnetron sputtering method ("B law" is called below) do not use the <sample production> RF exchange magnetron sputtering method ("A law" is called below) and RF, the sample of the Pd/Mg two-layer laminating structure (0.07Pa, 0.7Pa, and 7Pa) which carried out 3 level variate was produced for Ar gas pressure at the time of sputtering of Mg on the glass substrate. The sputtering conditions of Mg were set to DC0.05A, and when based on A law, they set RF power to 200W. Moreover, the sputtering conditions of Pd were set to 7x10 to 2 Pa Ar gas pressure, and DC0.1A, and when based on A law, they set RF power to 50W.
[0044] The <test evaluation approach> About each of the sample of a total of six sorts, it was made to move to a hydrogenation chamber, holding in a vacuum without putting the sample which formed membranes to atmospheric air, and hydrogenated by holding it under the ambient atmosphere of 373K by hydrogen-gas-pressure 0.1MPa for 24 hours.
[0045] Subsequently, the sample was held in the vacuum, the temperature up was carried out from a room temperature to 573K with the programming rate of 4 K/min, thermal temperature-programmed-desorption (TDS) analysis performed gas-evolution profile measurement, and temperature of the peak of the amount of gas evolutions was made into hydrogen desorption temperature.
[0046] Moreover, the crystal structure was analyzed by an X diffraction experiment and electron microscope observation about each of a sample.
[0047] <Test evaluation result> drawing 3 shows the relation between Ar gas pressure at the time of sputtering of Mg, and hydrogen desorption temperature. drawing 4 -- the configuration of the Pd/Mg two-layer laminating structure -- typical -- being shown -- (a) -- B -- what was made into 0.7Pa of Ar gas pressure by law, and (b) -- A -- what was made into 0.07Pa of Ar gas pressure by law, and (c) -- A -- it considers as 0.7Pa of Ar gas pressure by law.
[0048] According to drawing 3 , in [any] the case of Ar gas pressure, it turns out that the sample produced by A law has hydrogen desorption temperature lower than the sample produced by B law. moreover, A -- law and B -- it also turns out that hydrogen desorption temperature is low rather than the direction which set Ar gas pressure to 0.7Pa also sets to 0.07Pa or 7Pa the sample produced by which approach of law.
[0049] As shown in drawing 4 , the columnar crystal which carried out orientation also of any to Mg layer in the direction of thickness was checked, but it turns out that the column width is small, so that hydrogen desorption temperature becomes low. According to crystal structure analysis, in what was made into 0.7Pa of Ar gas pressure by A law, the column width of a columnar crystal was 100nm or less.
[0050] as mentioned above, the Pd/Mg two-layer laminating structure -- setting -- A -- by setting Ar gas pressure at the time of sputtering to 0.5-1.0Pa by law, while the nano columnar crystal structure where column width was small (100nm or less) was formed in Mg layer, it was checked that the hydrogen desorption temperature of the Pd/Mg two-layer laminating structure will become low.
[0051] (Test evaluation 2)

< sample production >A -- law and B -- by each approach of law, the sample of the Pd/Mg two-layer laminating structure (100nm, 200nm, 400nm, 600nm, and 800nm) which carried out 5 level variate was produced for the thickness of Mg layer on the glass substrate. The sputtering conditions of Mg were set to 7x10 to 1 Pa Ar gas pressure, and DC0.05A, and when based on A law, they set RF power to 200W. Moreover, the sputtering conditions of Pd were made into 7x10 to 2 Pa Ar gas pressure, DC0.1A, and 50nm of thickness, and when based on A law, they set RF power to 50W.

[0052] The <test evaluation approach> While searching for hydrogen desorption temperature like the test evaluation 1 about each sample of a total of ten sorts, 4-fold [in all] pole mass analysis was performed at the time of thermal temperature-programmed-desorption (TDS) analysis, and what measured the mass of the emitted hydrogen and converted it into the percentage to the weight of Mg layer was made into the amount of hydrogen desorption.

[0053] <Test evaluation result> drawing 5 shows the relation between the thickness of Mg layer, and a hydrogen burst size. Drawing 6 shows the relation between the thickness of Mg layer, and hydrogen desorption temperature.

[0054] according to drawing 5 -- A -- law and B -- also in the sample produced by which approach of law, the thickness of Mg layer becomes thick -- although it is alike, and it follows and the hydrogen burst size is falling -- A -- although produced by law, it turns out that a fall inclination [in / in the direction / 400nm or more in thickness] is small.

[0055] according to drawing 6 -- A -- law and B -- the sample produced by which approach of law is also known by that it is alike, and follow and hydrogen desorption temperature is falling [to which the thickness of Mg layer becomes thick].

[0056] (Test evaluation 3)

By <sample production> A law, the sample of the three layer laminating structure (25nm, 50nm, 100nm, 200nm, 400nm, 600nm, 800nm, and 1200nm) of Pd/Mg/Pd which carried out 8 level variate was produced for the thickness of Mg layer on the glass substrate. Moreover, the sample of the Pd/Mg two-layer laminating structure which set thickness of Mg layer to 1200nm was also produced by A law. Each sputtering condition of Mg and Pd presupposed that it is the same as that of the test evaluation 2.

[0057] The <test evaluation approach> Hydrogen desorption temperature and the amount of hydrogen desorption were calculated by the same approach as the test evaluation 2 about each sample of a total of nine sorts. Here, in the sample of the three layer laminating structure 400nm or more of Pd/Mg/Pd, the sample exfoliated from the glass substrate at the time of a hydrogen treating, and the thickness of Mg layer analyzed with the exfoliative sample simple substance.

[0058] Moreover, the crystal structure was analyzed by an X diffraction experiment and electron microscope observation about each of a sample.

[0059] <Test evaluation result> drawing 7 shows an example of the sample of the produced three layer laminating structure of Pd/Mg/Pd. Drawing 8 shows the example of a profile of thermal temperature-programmed-desorption (TDS) analysis of the hydrogenated three layer laminating structure of Pd/Mg/Pd (the ion current corresponds to a hydrogen burst size). Drawing 9 shows the relation between the thickness of Mg layer, and a hydrogen burst size. Drawing 10 shows the relation between the thickness of Mg layer, and hydrogen desorption temperature. In addition, the data in the test evaluation 2 are used by the data of the Mg/Pd two-layer laminating structure drawing 9 and among 10 except that whose thickness of Mg layer is 1200nm.

[0060] The sample of the three layer laminating structure of Pd/Mg/Pd has the composition that the laminating of Pd layer, Mg layer, and the Pd layer was carried out on the glass substrate at order as shown in drawing 7 , and crystal structure analysis showed that Mg layer had the columnar crystal structure with a column width of 10-50nm which carried out nano structuring. the front face of the substrate since the latter is Pd layer to the substrate of the former in the case of sputtering of Mg layer being a glass substrate as for the thing with smaller three layer laminating structure [of Pd/Mg/Pd] crystal size compared with the Pd/Mg two-layer laminating structure (column width of 100nm or less) of the test evaluation 1 -- it is presumed that the difference of description and a heat-conduction property influences.

[0061] According to drawing 8 , it turns out that the temperature from which the hydrogen burst size from Mg layer serves as a peak, i.e., hydrogen desorption temperature, has shifted to a low temperature side, so that the thickness of Mg layer becomes thick. Moreover, since the peak value is increasing so that the thickness of Mg layer becomes thick, it turns out that the amount of hydrogen which occlusion was carried out to Mg layer and emitted to it is increasing.
[0062] According to drawing 9 , in the case of the three layer laminating structure of Pd/Mg/Pd, the hydrogen burst size is maintaining the level more than 4.4 - 5.9 mass % and 4 mass % to the hydrogen burst size falling [the thickness of Mg layer] even to 2.6 mass % from 5.7 mass % in the range to 800nm in the case of the Pd/Mg two-layer laminating structure. By the Pd/Mg two-layer laminating structure, since a hydrogen atom will not permeate to Mg layer core if the thickness of Mg layer is set to 600nm or more, the occlusion of hydrogen becomes imperfect, and this is considered to have led to reduction of the amount of hydrogen desorption. By the three layer laminating structure of Pd/Mg/Pd, since Pd layer is in the vertical both sides of Mg layer, even if Mg layer becomes thick, a hydrogen atom is considered to be what permeates promptly to Mg layer core. However, also in the three layer laminating structure of Pd/Mg/Pd, if the thickness of Mg layer is set to 1200nm, the amount of hydrogen desorption will be low, and it is thought that this is because the occlusion of hydrogen became imperfect since a hydrogen atom did not permeate to Mg layer core.
[0063] According to drawing 10 , hydrogen desorption temperature has low-temperature-ized the three layer laminating structure of Pd/Mg/Pd, and the Pd/Mg two-layer laminating structure as the thickness of Mg layer becomes thick, and former one is known by that hydrogen desorption temperature is lower than the latter when the thickness of Mg layer is the same. Specifically, the thickness of Mg layer is falling even to 90 degrees C in 800nm or more at the latter to hydrogen desorption temperature falling [the thickness of Mg layer] to about 110 degrees C in 800nm or more by the former. Moreover, consideration of the result of the test evaluation 1 expects that hydrogen desorption temperature becomes low in the columnar crystal structure with smaller column width which carried out nano structuring.
[0064] By the three layer laminating structure of Pd/Mg/Pd, although the sample exfoliated [the thickness of Mg layer] from the glass substrate in 400nm or more at the time of hydrogenation, a touch area with the hydrogen gas of Pd layer will be greatly secured by this, and it is thought that it was connected with the bottom of maintenance of the high amount of hydrogen desorption and the low temperature of hydrogen desorption temperature. That is, a substrate is required in the phase which produces the laminating structure by the sputtering method etc. It is thought that a substrate is not required in case it is actually used as hydrogen absorption material, and the direction which does not have a substrate rather is excellent in hydrogen absorption and the emission characteristic (however, in case it is used as a negative electrode of a rechargeable battery). If a laminated structure is made to form in substrates, such as nickel, it can be used as a negative electrode as it is, and a manufacture process can be simplified.
[0065] As mentioned above, the three layer laminating structure of Pd/Mg/Pd was excellent in the property of reducing hydrogen desorption temperature, compared with the Pd/Mg two-layer laminating structure, maintaining the high amount of hydrogen desorption, and especially, when the thickness of Mg layer was thick (400-800nm), it was checked that it becomes remarkable.
[0066] (Test evaluation 4)
The thickness of a <sample production> Mg layer is fixed to 200nm. By A law The three layer laminating structure of Pd/Mg/Pd (200nm in whole Mg layer thickness), The five layer laminating structure of Pd/Mg/Pd/Mg/Pd (400nm in whole Mg layer thickness), Pd/Mg/-- The seven layer laminating structure of /Pd (600nm in whole Mg layer thickness), The sample of the nine-layer laminating structure (800nm in whole Mg layer thickness), the 11-layer laminating structure (1000nm in whole Mg layer thickness), and the 13-layer laminating structure (1200nm in whole Mg layer thickness) was produced on the glass substrate, respectively. Each sputtering condition of Mg and Pd presupposed that it is the same as that of the test evaluation 2.
[0067] The <test evaluation approach> Hydrogen desorption temperature and the amount of hydrogen

desorption were calculated by the same approach as the test evaluation 2 about each sample of a total of six sorts.
[0068]

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is the mimetic diagram showing the structure of the hydrogen absorption laminating structure concerning the operation gestalt of this invention.
[Drawing 2] It is the block diagram of RF exchange magnetron sputtering system.
[Drawing 3] It is the graphical representation showing the relation between Ar gas pressure at the time of sputtering of Mg, and hydrogen desorption temperature.
[Drawing 4] It is the mimetic diagram showing the configuration of the Pd/Mg two-layer laminating structure.
[Drawing 5] It is the graphical representation showing the relation of the thickness of Mg layer and the amount of hydrogen desorption in the test evaluation 2.
[Drawing 6] It is the graphical representation showing the relation of the thickness of Mg layer and hydrogen desorption temperature in the test evaluation 2.
[Drawing 7] It is the mimetic diagram showing the configuration of the three layer laminating structure of Pd/Mg/Pd.
[Drawing 8] It is the graphical representation showing the example of a profile of thermal temperature-programmed-desorption (TDS) analysis of the hydrogenated three layer laminating structure of Pd/Mg/Pd.
[Drawing 9] It is the graphical representation showing the relation of the thickness of Mg layer and the amount of hydrogen desorption in the test evaluation 3.
[Drawing 10] It is the graphical representation showing the relation of the thickness of Mg layer and hydrogen desorption temperature in the test evaluation 3.
[Drawing 11] It is the graphical representation showing the relation of the whole Mg layer thickness and the amount of hydrogen desorption in the test evaluation 4.
[Drawing 12] It is the graphical representation showing the relation of the whole Mg layer thickness and hydrogen desorption temperature in the test evaluation 4.
[Drawing 13] It is the graphical representation showing the relation between the light wave length of the three layer laminating structure of Pd/Mg/Pd, and light transmittance.
[Description of Notations]
1 Hydrogen Absorption Laminating Structure
2 Hydrogen Absorption Layer
2a Nano columnar crystal
3 Hydrogen Transfer Layer

[Translation done.]

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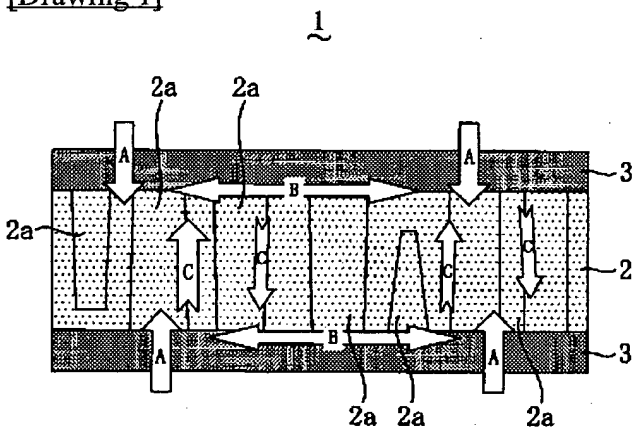
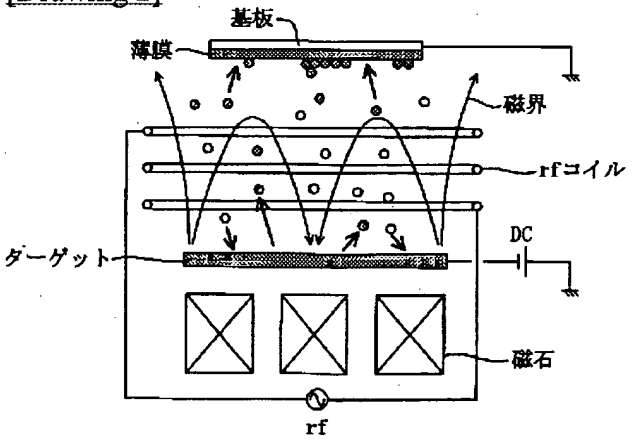
DRAWINGS

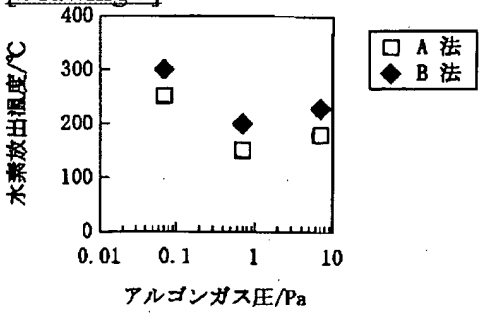
[Drawing 1]



[Drawing 2]

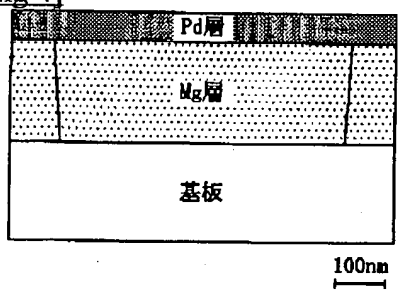


[Drawing 3]



[Drawing 4]

(a)



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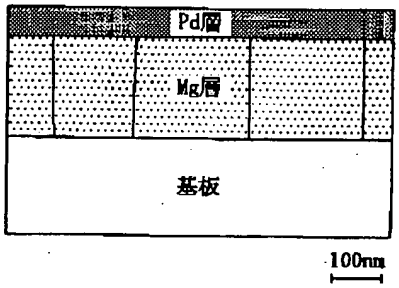

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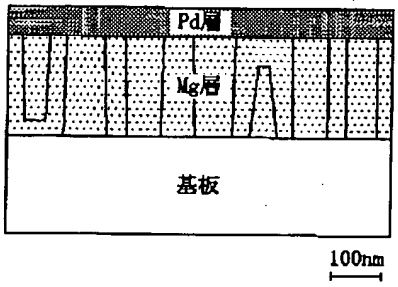
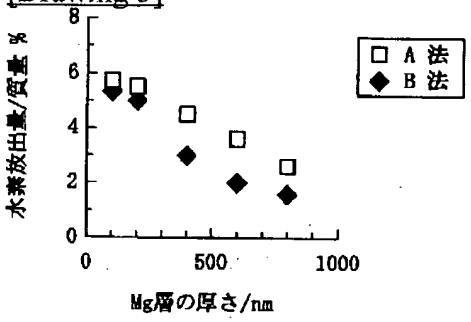
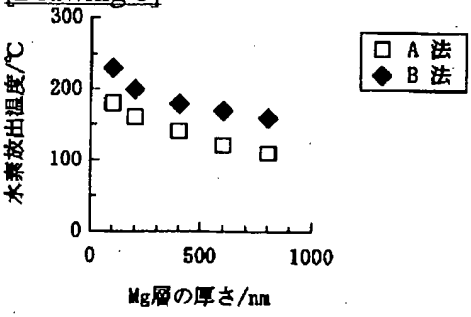


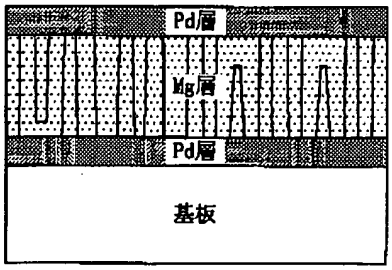
[Drawing 5]



[Drawing 6]

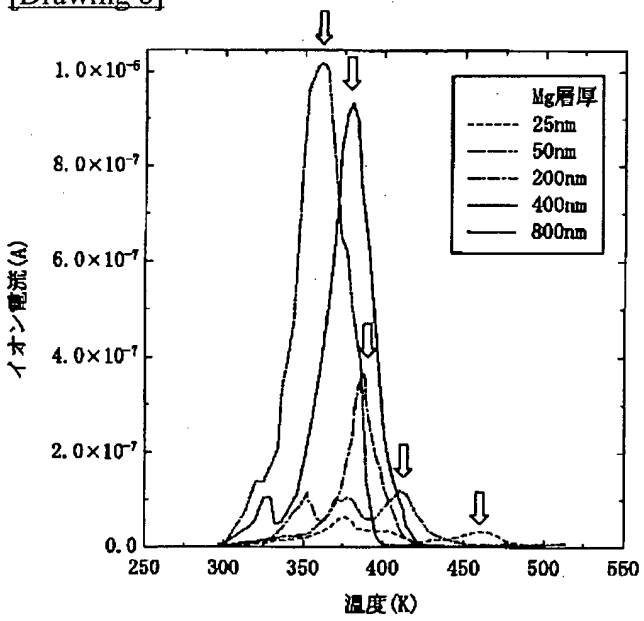


[Drawing 7]

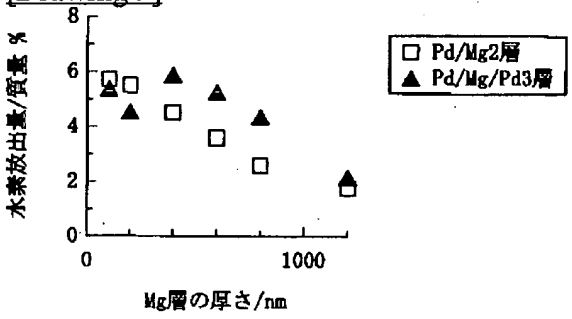


100nm

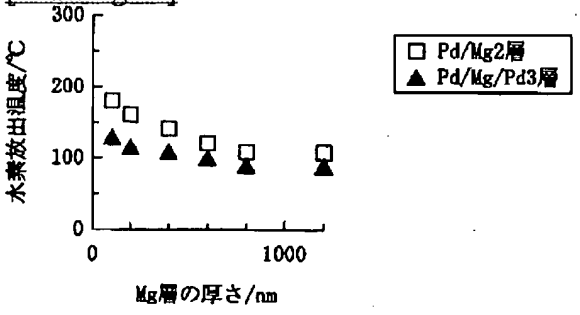
[Drawing 8]



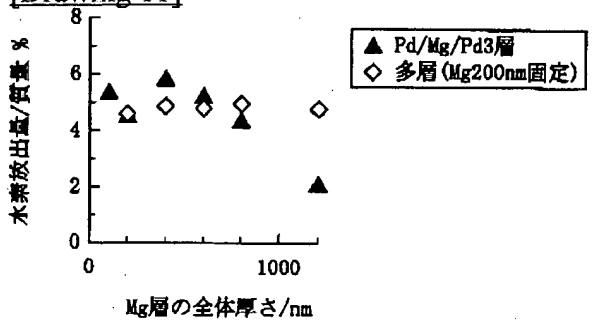
[Drawing 9]



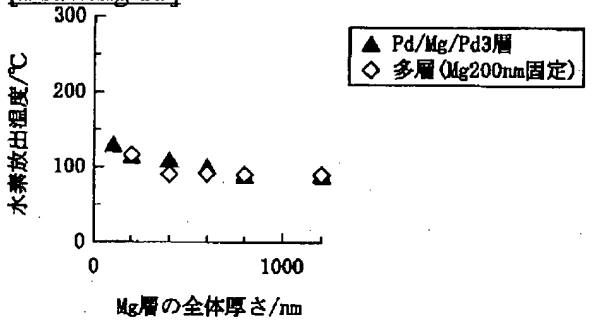
[Drawing 10]



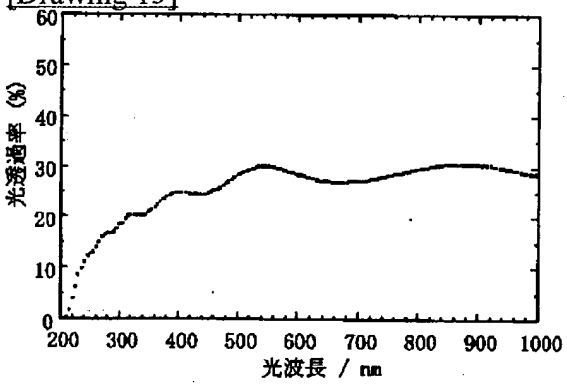
[Drawing 11]



[Drawing 12]



[Drawing 13]



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